CANADA

SIMPLIFIED APPARATUS AND TECHNIQUE FOR THE DETERMINATION OF CRYSTAL ORIENTATION BY ION BOMBARDMENT

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SIMPLIFIED APPARATUS AND TECHNIQUE FOR THE DETERMINATION OF CRYSTAL ORIENTATION BY ION BOMBARDMENT *

R. L. Cunningham ** and Joyce Ng-Yelim ***

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ABSTRACT

The ion bombardment camera, designed specifically for orientation determinations, is machined from clear acrylic plastic and incorporates an ion source of the glow discharge type. A plastic orienting sphere, illuminated from within, has engraved on it the low index directions of cubic crystals. Hemispherical collectors, bearing ejection patterns, are positioned on the sphere and the orientation of the crystals may then be immediately observed.

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Direction des mines

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UN APPAREIL ET UNE TECHNIQUE SIMPLIFIÉS POUR DÉTERMINER L'ORIENTATION DES CRISTAUX PAR BOMBARDEMENT IONIQUE*

R. L. Cunningham** et Joyce Ng-Yelim***

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RÉSUMÉ

La chambre à bombardement ionique, conçue spécialement en vue de déterminer l'orientation, est fabriquée en plastique transparent (résine acrylique). Elle comprend une source ionique du type à décharge luminescente. Les indices simples des cristaux cubiques sont gravés sur une sphère d'orientation en plastique, illuminée de l'intérieur. Des collecteurs hémisphériques, sur lesquels les diagrammes d'émission secondaire sont enrégistrés, sont en position sur la sphère, et il est ainsi possible d'observer immédiatement l'orientation des cristaux.

*Cette étude a paru dans "The Review of Scientific Instruments", vol. 36, n° 1, Janvier 1965, pp. 54-56.

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INTRODUCTION

Crystal orientation is usually determined by the use of X-ray and electron diffraction methods or by the observation of planes developed during growth, etching or cleaving. In 1955 Wehner (1) reported the directional ejection of atoms caused by the ion bombardment of single crystals of bcc alpha-iron and tungsten, and fcc silver and copper, and suggested that the patterns so formed might make possible approximate orientation determinations. Nelson [2], by collecting the ejected atoms on flat plates, qualitatively analysed the preferred orientation of foils of the fcc metals silver, gold, copper, palladium and rhodium. These were prepared both by evaporation on salt substrates and by rolling and annealing. Southern, Willis and Robinson [3] stated that patterns obtained from copper and collected on flat plates could be used to determine orientations about as accurately as by the use of Laue back-reflection photographs, "at least near (001)".

In a study of ejection from single crystals of aluminum, Cunningham, Gow and Ng-Yelim [4] obtained well defined patterns on hemispherical collectors which pointed to the feasibility of using the method for orientation determinations provided that sufficiently short exposure times could be obtained.

Recently, the present authors [5] successfully applied the method to the orientation of the bcc metals tungsten, iron and beta-brass and the fcc metals silver, copper and aluminum over a wide range of orientations, using apparatus designed for general ion bombardment studies, and estimated the maximum error to be about 3°. Exposure times, using 8 KeV argon ions at a specimen current of 100-200 microamperes, ranged from 15 seconds for silver to 5 minutes for aluminum. A complete orientation determination takes 20 minutes or less since no photographic processing is involved.

The utility of the method having thus been established for many common metals, new and simplified apparatus and technique have been developed for orientation determinations. The apparatus is very inexpensive to construct and should prove advantageous where orientations of single crystals are required of those metals suitable for bombardment in the medium energy range. In addition to the camera, a power supply and a vacuum system with a background pressure of 0.01 micron or better are required. A cold trap is used to prevent the arrival of pump oil at the specimen surface where it would be rapidly polymerized by the impinging ions. Mass spectrometer grade argon is used for reactive metals.

THE CAMERA

The ion bombardment camera is shown in Figures 1 and 2. It is machined from $3\frac{1}{2}$ in. diameter rod of clear acrylic plastic (Plexiglas). The ion source being of the glow discharge type has a large output. The glow discharge region and the bombardment chamber are separated by the thin, easily replaceable, 0.25 mm aluminum cathode (A) in contact with the sealing "O" ring (B) and pierced with a central hole of about 0.5 mm diameter. The hole at the upper end of the cylindrical aluminum anode (C) is slightly tapered to accommodate a short length of polyethylene tubing used as a vacuum seal between the drawn-down glass capillary and the anode. The supply gas, controlled by a pressure regulator and passing through this capillary, enters the glow discharge region through a 1 mm axial hole at the lower end of the anode, the sharp edge of which helps to concentrate the glow discharge opposite the hole in the cathode for maximum output; the outer edge of the anode is slightly rounded for the same reason. To prevent attack on the plastic near the glow discharge, the glass sleeve (D) surrounds the anode and is supported by the "O" ring (E). It is necessary that this sleeve contact the plastic at its upper end and the cathode at its lower end.

The anode-cathode distance is adjusted without rotation of the anode, by turning a threaded cover which supports it. The anode carries the copper cooling disc (F) which eliminates the possibility of the anode "O" ring (G) becoming overheated. Air-cooling of the copper disc is achieved by a series of holes in the cover. High voltage is supplied to the anode through its cooling disc by contact with the anchor pin (H) used to prevent the anode assembly from rotating. Electrical contact with the anode is avoided by making the upper gas input fitting (I) of plastic.

The pressure within the glow discharge is necessarily some tens of microns while the pressure in the bombardment chamber must be sufficiently low so that an ejected atom has a high probability of reaching the collector without collision with gas molecules, viz., a micron or less. The pressure differential is obtained by rapid pumping of the system so that the necessary pressure drop along the hole in the cathode is obtained.

Since it is not possible to rapidly pump down the glow discharge region through the small cathode hole, the cathode is temporarily held away from the sealing "O" ring (B), by pressing on the plastic fitting (I) at the upper end of the anode for a short period. The spring loaded copper disc (J) returns the cathode to its normal position when the force on the anode is released. This procedure is necessary when preparing to bombard aluminum crystals since directional ejection cannot occur in the presence of an oxide film.

The bombardment chamber contains the single crystal specimen (K), which may be as large as 18 x 18 x 6 mm, the specimen holder (L), the hemispherical collector (M), its retaining band (N) and the ring (O), and the supporting framework (P), which also serves to ground the cathode. All but the latter are insulated from ground by bushings (Q). This arrangement permits monitoring of the specimen current with a microammeter connected to ground through the shaft of the knob (R), whose primary purpose is to permit tilting of the assembly for inspection of the pattern at any time during bombardment.

THE COLLECTOR

The use of hemispheres, with the specimen at the centre of curvature, ensures that all ejected material is collected. Since the ejected atoms arrive in directions normal to the collector, an accurate estimation of the centre of gravity of each spot can be made. These are then carefully marked with pin holes. Spots from some metals tend to fade and in these cases the pin holes also ensure a permanent record. Hemispheres from ping-pong balls were chosen because they are easily cut, they are white, and of fairly uniform size. The hole for the input beam also serves as a marker for the pole of the bombarded surface and may be accurately punched at the top of the hemisphere by placing it in a suitable jig. The collector is protected from the ion beam during inspection periods by the aluminum band (N).

THE ORIENTING SPHERE

Since ejection is along crystallographic directions, all patterns formed on hemispherical collectors are suitable for the direct production of stereographic projections by an optical device (5). However, for cubic metals, a much simpler and quicker method is to place the hemispherical collector over a close fitting transparent plastic sphere (Figure 3).

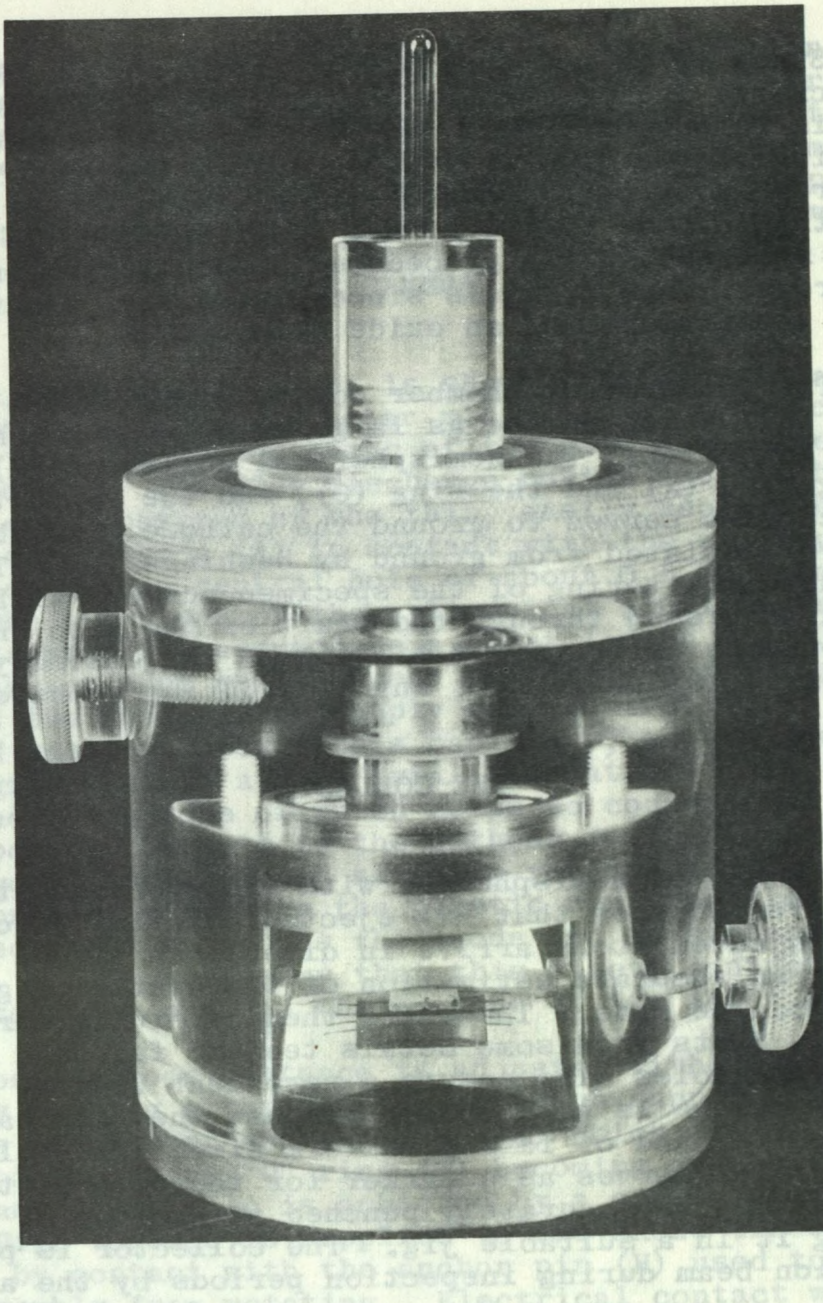

Figure 1. Camera with specimen holder slightly tilted to show single crystal in place.

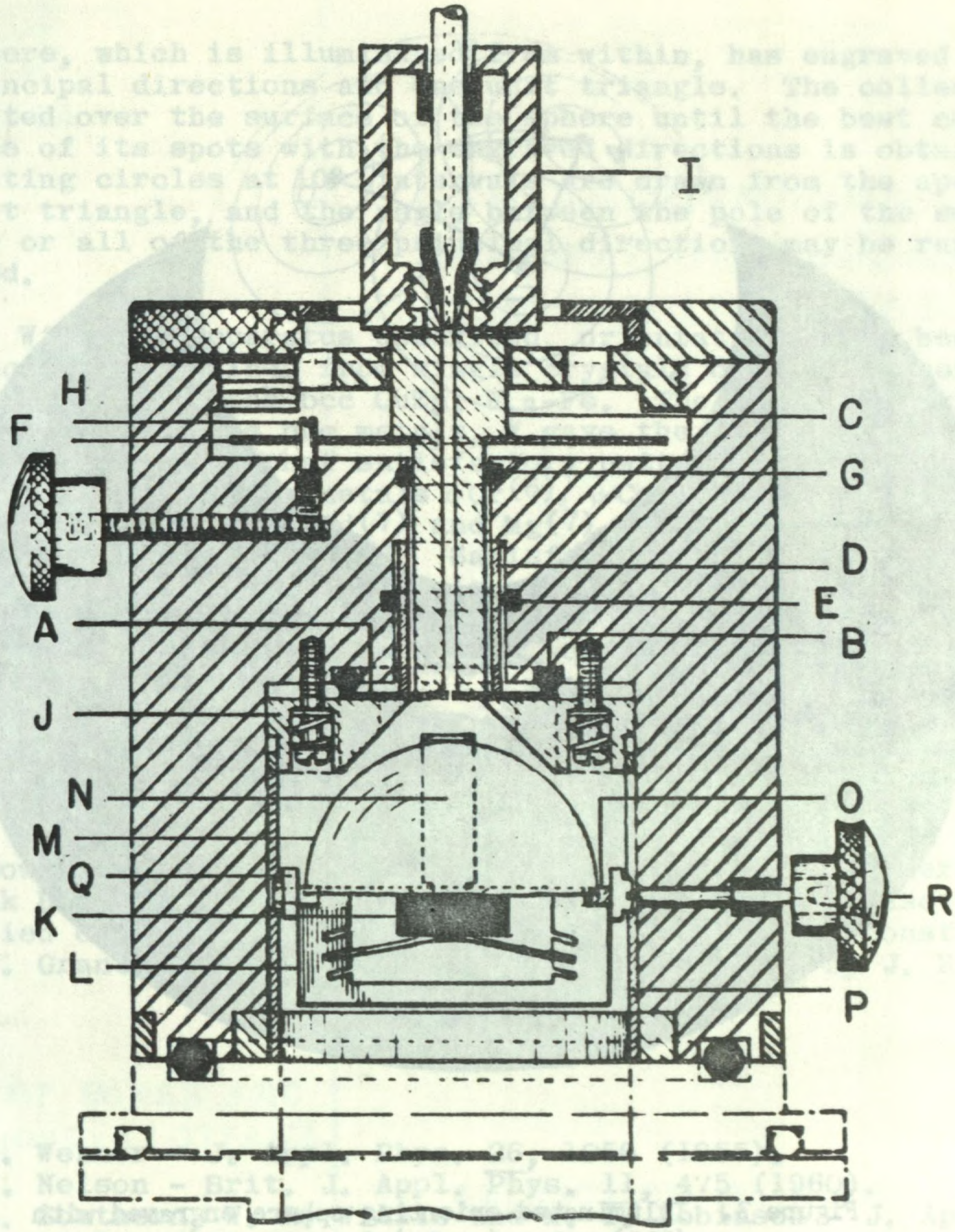

Figure 2. Cross section of ion bombardment camera.

A - aluminum cathode
B - cathode sealing "O" ring
C - aluminum anode
D - glass sleeve
E - supporting "O" ring
F - copper cooling disc
G - anode sealing "O" ring
H - anchor pin
I - plastic fitting
J - spring loaded copper disc
K - single crystal specimen
L - specimen holder
M - hemispherical collector
N - aluminum retaining band
O - collector ring
P - supporting framework
Q - insulating bushings
R - tilting knob

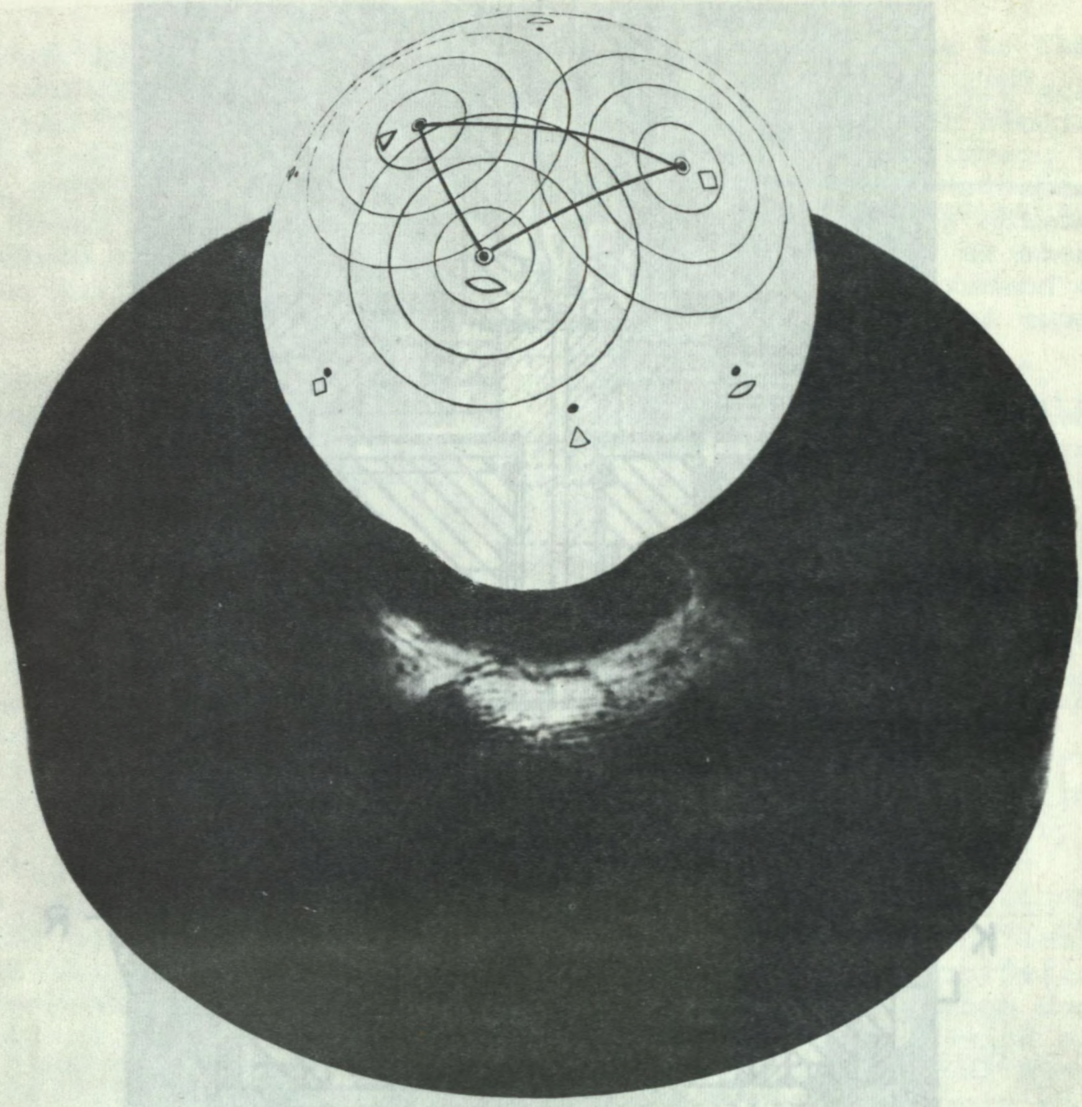

Figure 3. Illuminated orienting sphere engraved with (110) poles (ovals) for orientation of face centered cubic metals, and with (100) and (111) poles (squares and triangles respectively) for body centered cubic metals (5).

The sphere, which is illuminated from within, has engraved on it the principal directions and one unit triangle. The collector is shifted over the surface of the sphere until the best correspondence of its spots with the engraved directions is obtained. Calibrating circles at 10° intervals are drawn from the apexes of the unit triangle, and the angle between the pole of the surface and any or all of the three principal directions may be rapidly observed.

With the apparatus described, orientations have been satisfactorily obtained from single crystals of the fcc metals Au, Ag, Cu, Al, Pb and the bcc CuZn, W, α-Fe. The fcc metals gave the best patterns; of the bcc metals, W gave the best and Fe the poorest. Known data indicates that satisfactory patterns have also been obtained from the cubic metals α-Cr[6], β-Co[6], Pd[2], Rh[2] and the hexagonal metals Zn[7], Cd[7] and Mg[7] when bombarding energies of several keV were employed. Satisfactory patterns were not obtained from our single crystals of niobium, tantalum and vanadium although Anderson [8] has reported directional ejection from these bcc metals at much lower bombarding ion energies.

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